2547-101

WAR DEPARTMENT INTELLIGENCE

JAPANESE INSECTICIDE

27 MARCH 1947

U.S. Army

REPRODUCED BY 5250TH TECHNICAL INTELLIGENCE COMPANY
APO 500

5250th TECHNICAL INTELLIGENCE COMPANY
WDI DIV, G-2 GHQ FEC

APO 500
31 March 1947

SUBJECT: Report of Investigation - Japanese Insecticide (Supplementary Report)

TO : Director, WDI DIV

1. AUTHORITY: G-2 I.O.M., from Director, WDI DIV to 5250th TIC, subject: "Japanese Insecticide", dated 7 February 1947.

2. REFERENCES:

a. Letter, War Department General Staff, Intelligence Division, MID File No. 070, subject: "Japanese Insecticide", dated 24 January 1947 (Control No. MD-610).

b. Report of Investigation - Japanese Insecticide, by 5250th Technical Intelligence Company, dated 18 February 1947. This report forwarded all data then available in reply torequest for information from War Department (reference a) and recommended further investigation along indicated lines.

3. DETAILS OF INVESTIGATION:

a. Investigators visited the Central Laboratory of the Kanegafuchi Spinning Co., Ltd., Osaka 12 March 1947. Yoshio MIKAMI, In charge of the chemical branch of the company, was interviewed and disclosed that he had worked for six months with Professor SHISHIDO, Kyoto Imperial University, on research of insecticides, and that the company and SHISHIDO has jointly applied for a patent on the manufacture of 'KANEBO BHC', andinsect powder consisting of 10% benezene hexachloride and 90% talc or clay. Copy of Translation of Patent Application received from MIKAMI is inclosed (Incl 1).

MIKAMI stated that the company had manufactured two tons of the product; one ton, packed in 20 kg. paper bags, at their Osaka pilot plant in July 1946, and the other ton, packed in cardboard cans (50 gms) at their TAKASAGO Works in December 1946. He further stated that the TAKASAGO Works, located in Takasago-cho, Hyogo-ken, has a monthly capacity of ten tons of 10% BHC, but is not now producing pending receipt of permission from the Imperial Japanese Government and SCAP.

When asked whether or not other applications has been filed covering the manufacture of Benzene Hexachloride, MIKAMI stated that he has seen a sample of an insecticide powder at the Tokyo patent office, but did not know by whom it has been submitted, and that he knew that Mitsubishi K.K. has submitted a sample of an insecticide similiar to BHC to Kyoto University for a study.

MIKAMI gave the investigator a mimeographed sheet which included extracts from various periodicals (Incl 2) on the subject of insecticides. He stated that these articles has prompted Professor SHISHIDO's study of insecticides. MIKAMI also provided a sample of 100% BHC (Incl 3), a sample of 10% BHC (Incl 4) and notes of the study he had made. A copy of translation of these notes is inclosed (Incl 5).

b. During a routine visit to the Central Laboratory of the Kanegafuchi Co, Osaka, in March 1947, for surveillance of their research activities, investigators received a copy of the company's semi-annual report to SCAP. Extracts pertinent to research on insecticides are inclosed (Incl 6).

c. Investigators interviewed Professor Keiichi SHISHIDO, Department of Industrial Chemistry, Faculty of Engineering, Kyoto

Imperial University in Kyoto 11 March 1947. SHISHIDO stated that he had become interested in insecticides after having read extracts from various American and British periodicals (he produced the same mimeographed document, Incl 2, which was later received from MIKAMI) and that he had 'invented' benzene hexachloride and has applied for a patent in conjunction with the Kanegafuchi Spinning Co., Ltd., Osaka. He gave the investigators a sample of 100% BHC prepared by himself (Incl 7). He admitted that he was familiar with the product 'GAMMEXANE', which is covered by British Patents, but that he did not know whether or not it was the same isomer of benzene hexachloride as his product. He indicated that he had written an article comparing BHC with DDT for the November-December issue of 'KAGAKU-GOHO' (Copies of this periodical were obtained in Tokyo and a translation of his article is inclosed (Incl 8). SHISHIDO denied any knowledge of WATANABE, the chemist who had been reported as having invented BHC in Yokohama.

d. Investigators interviewed Keizo NOMOTO of the Imperial Japanese Patent Office on 27 March 1947. NOMOTO stated that the patent application (Incl 1) was under examination at that office. The Application was scanned and was sent back to the applicant because it contained ambiguous statements and imperfect points. The applicant was instructed to resubmit an application with a report of the results of experiments and to clarify the ambiguous points. This reapplication had not been received by the Patent Office on the date of this interview.

4. CONCLUSIONS:

a. Benzene Hexachloride is not a new product in the field of insecticides. Kanegafuchi Spinning Co. Ltd. has made application for permission to manufacture and sell insecticide (Incl 9) and plans to produce 10% BHC commercially if permission is granted by the Japanese Government and SCAP.

b. Further investigation at this time is not indicated.

5. RECOMMENDATIONS:

It is recommended that this report, with inclosures, be forwarded to Director of Intelligence, War Department General Staff, Washington 25, D.C.

RALPH L FOSTER
Lt Col Cav
Commanding

9 Inclosures:

Incl 1: Copy of translation of Patent Application submitted by Kanegafuchi Spinning Co.
Incl 2: Copy of mimeographed extracts of periodicals.
*Incl 3: Sample of 100% BHC (Kanegafuchi)
*Incl 4: Sample of 10% BHC (Kanegafuchi)
Incl 5: Translation of notes on a study by MIKAMI.
Incl 6: Extracts from Semi-annual Report, Kanegafuchi Spinning Co.
*Incl 7: Sample of 100% BHC (SHISHIDO)
Incl 8: Translation of article (SHISHIDO) from 'KAGAKU-GOHO'.
Incl 9: Copy of translation of Application for Permission to Manufacture and Sell Insecticide.

* With original (MID, WD) copy only.

I. Discription of the Patent Application.

Patent Claimant: KANEGAFUCHI SPINNING CO., Ltd.
No. 347, OI-YORICHO, SHINAGAWA Ward, TOKYO.

Inventors: KEIICHI SHISHIDO
No. 36, IZUMIKAWA MACHI, SHIMOGAMO, SAKYO Ward, KYOTO.
YOSHIO MIKAMI
No. 398, 1 Chome, YOSHIDA Cho, HYOGO Ward, KOBE.

Manufactoring technique of crystalling chlorinated compounds from aromatic hydrocarbons or chlorinated aromatic hydrocarbons.

Outline of the nature and objectives of the patent.

This discovery will enable the manufacture of a crystalline chlorinated compound from aromatic hydrocarbons or chlorinated aromatic aromatic hydrocarbons by gradually adding a dilute alkaline catalyser during the process of chlorination at a temperature lower than ten (10) degrees Centigrade. The objective is to get a crystalline isomer of these compounds having strong insecticide properties in a convenient form.

Detailed Discription of the Invention.

When aromatic hydrocarbons or its chlorides are chlorinated in a dilute alkaline solution in the presence of light, a chlorinated compound is obtained. For example;

Benzenhexachloride derived from benzene.

Mono chloro-benzenhexachloride from monochlorobehzene.

Ortho or para-dichloro-benzen hexachloride derived from ortho or paradichlorobenzene respectively.

Each example is obtained in the specific state of its isomeric compound.

Among these examples, benzenhexachloride is well known for its insectidide properties. However, the researchers have discovered that the latter two examples have strong insecticide properties and at the same time sustain insecticide powers much longer, with less disagreeable odor, because of its involatile nature.

The time necessary for the chemical reaction is long while the amount obtained is far from satisfactory under the present method of manufactoring. The yield of the crystalling isomers, which is the most convenient form for an insecticide, is very small.

After repeated laboratory tests, the researchers discovered that this chemical reaction can be accelerated if it is performed at temperature less than ten (10) degrees centigrade. The yield is also increased.

The catalyser, a dilute alkaline solution, is added gradually during the chemical reaction. Not all at once. Furthermore, by combining the two above processes, the most effective results can be obtained. The yield of the crystalling compound is larger and is crystallized out of the solution in the original process, the precipitate is purified by steam distillation, etc. However, this new technique elimates this process and the crystalling compound is seperated by filtering.

By standard procedures, approximately 17 parts of crystalling benzen hexachloride is obtained from 100 parts of benzene at room temperature of about 20° Centigrade after seven hours of treatment.

By the new procedure, 140 parts of crystalling benzenhexachloride can be obtained at a temperature of three to five degrees Centigrade in three hours.

Example I.

To 100 parts of benzene, add 10 parts of a one percent solution of sodium hydroxide (Na HO) as a catalyzer. During the first part of the process, add only 1/3 of this amount and let it stand. This solution is then cooled to a temperature of about three to five degrees Centigrade. When chlorine is added to this solution, the benzene reacts with the chlorine and increases its specific gravity. The benzen hexachloride starts to precipitate from the solution in a crystalling state. At this instant, the catalyser is gradually added to push. The chemical reaction along after the benzen hexachloride has been crystallized out as much as possible, it is filtered and washed with water. This yield consists of various compounds of certain geometrical isomers having the formula $C_6H_6Cl_6$ and the amount is 140 parts.

Example II.

To 100 parts of monochloro benzene, add three parts of a one percent solution of sodium hydroxide (NaOH). This mixture is cooled to a temperature of about five degrees Centigrade and chlorine is added. When crystals of monochloro benzene hexachloride begin to form, seven parts of a ten percent (0.10) solution of sodium hydroxide is added gradually as a catalyser aid the chemical reaction of the chlorine. The greater part of the monochlorobenzene is crystallized out as an isomeric compound of monochloro benzenhexachloride, $C_6H_5Cl_7$, the yield of which is 70 parts.

Example III.

When 100 parts of industrial ortho-dichlorobenzene is treated with ten parts of a one percent (0.01) solution of sodium hydroxide as a catayser, under the same conditions as in Example I, 60 parts of ortho-dichlorobenzenhexachloride $C_6H_4Cl_8$ are obtained.

Example IV.

To 100 parts of para-dichlorobenzene dissolved in carbon tetrachloride ($C\ Cl_4$) add 10 parts of a one percent (0.01) solution of sodium heydroxide at a temperature of about three degrees Centigrade. Add chlorine. At first, add only 1/3 of the sodium hydroxide solution and after the mixture is saturated with chlorine, the remaining sodium hydroxide solution is added. The carbon tetrachloride layer is separated from the mixture and by means of distillation the carbon tetrachloride is eliminated so that 55 parts of paradichlorobenzenhexachloride, $C_6H_4Cl_8$ is obtained.

The scope of the patent application.

The patent application will cover the above mentioned production technique and the following:

While producing chlorinated compounds from aromatic hydrocarbons or chlorinated aromatic hydrocarbons by means of chlorination, the dilute alkaline catalyser is introduced at a tempertuer lower than ten (10) degrees centigrade. Also covered is the part on the special process of obtaining crystalline chlorinated compounds by gradually adding the catalyser as the chemical reaction of the chlorine progresses.

Bad News for Insects

-- Find Super-DDT

(Popular Mechanics, 84, No. 3, p. 10; Sept. 1945)

Discovery of a synthetic compound deadlier to insects than DDT is reported by British chemists. Called "Gammexane" or 666, the super-DDT is chemically the gamma isomer of benzene hexachloride, and is not particularly close to DDT in structural chemistry. Tests showed it to be the deadliest weevil poison ever tested by the British. Half the concentration required in a DDT solution killed flies. Length of its effectiveness is unknown.

Gammexane Said to Be More Powerful than DDT (Chem. & Eng. News) 23 849 (1945)

In the Hurter Memorial Lecture to the Society of Chemical Industry in Liverpool, Roland Slade, research controller of Imperial Chemical Industries, revealed the discovery of a new insecticide called Gammexane or 666. The new substance has been found highly poisonous to many insects, including locusts, wireworms, caterpillers, and larvae of the yellow fever mosquito, the grain weevil, and the apple blossom weevil. It is said to be five times more powerful in insecticidal effect than DDT. The research workwas carried out at the I. C. I's biological laboratory at Hawthorndale, Berks.

Bane for Locusts

(Newsweek, June 10, 1946)

Against the horde of locusts, Sardinian peasants fought desperately with brooms and rakes, with bonfires and scalding water. But by May 7 the advancing insects had swarmed overland in columns four layers deep, covering an area $2\frac{1}{2}$ miles wide by 30 miles long. "Unless the menace is checked by July," said Prof. Antonic Melis, director of the anti-locust service, "the locusts may completely extinguish plant and vegetable life on the island, rendering life impossible for man and animals."

Sodium arsenate is the standard chemical preparation for destroying the locust. But on searching both British and Continental sources, UNRRA procurement officers found none available for immediate shipment to Sardinia. Instead, on May 16, they sent from London by five chartered planes 10 tons of gammexane (NEWSWEEK, March 18) a new synthetic insecticide already successful against locust plagues in Iran, Iraq, and Kenya, and said to be 150 times more effective than sodium arsenite.

The 10-ton air shipment of gammexane, plus an additional 15 tons procured by UNRRA for delivery to Sardinia within the next weeks, may determine whether science has finally gained controllover the ancient scourge of locust migration.

Brother to DDT

(Newsweek, March 18, 1946)

The new British insecticide, benzene-hexacloride, is more toxic to flies and cockroaches than DDT. In agriculture tests it has proved superior to the American pest killer against such major nuisances as aphids, grasshoppers wireworms, and several cotton insects including the boll weevil.

But the English-developed chemical does not replace DDT, Dr. W. H. Tisdale, director of the Du Pont Pest Control Research Laboratory reported last week. Because of its disagreeable, pugent odor, the use of benzene hexachloride is limited. Experiments also indicate that it is more likely than DDT to damage foliage, especially such tender truck crops as lettuce and spinach.

Incl 2 to report of Investigation 5250th TIC 27 Mar '47

Deadlier Insecticide

(Time, June 24, 1946)

U. S. entomologists last week made a preliminary report on another sensational British insect killer, Gammexane claimed to be five times as deadly as DDT (Time, May 28, 1945). It has an unpleasant naphthalene smell, lacks DDT's lasting effect. It is particularly potent against cockroaches, proved effective in checking a locust plague in Sardinia this spring and has shown promise against the cotton boll weevil. But in the sunny U. S. climate it has been generally less lethal than in foggy Britain.

ANNUAL REVIEW OF BIOCHEMISTRY VOL XV 1946

Another organic chloride of much promise as an insecticide is hexachloro-cyclohexane, $C_6H_6Cl_6$, to which the name 666 has been given (146). This substance has been known as a chemical for a long time and is remarkable for the large number of possible isomers. Of these, four have been isolated and their structures determined with some certainty. It has been found that the so-called gamma isomer accounts for most of the toxicity of the crude product. Hence the term Gammexane has been proposed for this isomer. Its toxicity to house flies is very high in kerosene sprays nearly ten times that of DDT (147).

A special advantage seems to be high killing power for mites which are not affected by DDT (148). Exceptional stability to light and heat indicate that 666 will have a long period of effectiveness but this circumstance may also lead to complications if it is used on edible products.

(146) Slade R., Chemistry and Industry 314-19(1945)
(147) Gersdorff W.A. and Mc Gooran E.R., Soap 21,117,121 (1945)
(148) Tayler E.L., Nature 155 393-94 (1945)

INSECTICIDES AND RODENTICIDES

Frederick S. Philips

Section of Pharmacology, Medical Division,
Chemical Warfare Service, Edgewood Arsenal, Maryland.

Hexachlorocyclohexane

Of the many compounds which were surveyed as potential insecticides during the course of the war only 1, 2, 3, 4, 5, 6 --hexachloro-cyclohexane has proved to equal and in some instances surpass the toxicity of DDT for various species of insects. The compound developed through the effects of British investigators is commonly referred to as 666, to symbolize the empirical formula, $C_6 H_6 Cl_6$. Technical preparation of 666 are composed of varying proportions of 4 space isomers which differ structurally in the special arrangement of the chlorine and hydrogen atoms about the six-membered ring of carbon atoms. The 4 isomers also differ widely in their insecticidal actions. Thus, the gamma isomer, known as gammexane is many hundred times more toxic to insects than any of the other isomers. (26, 27)

Likewise the marked difference in activity of the isomers has been noted in mammalian species. Following oral administration gammexane is at least 5 times more toxic than the other isomers (27, 4). Similarly rabbits survive the intravenous administration of 100 mg./kg. of the delta isomer but succumb to 5 mg./kg. of gammexane (28, 29). While gammexane is a potent convulsant, the beta and delta isomers appear to be depressant eliciting marked flaccid paralysis in rabbits without concomitant signs of central stimulation (29). Moreover, the prior administration of the delta isomer can largely antagonize the convulsant actions of stravenous lethal doses of gammexane and thereby effect a severalfold reduction in its toxicity. (29).

(26) Tanner, C. C. By Communication, 1944
(27) Slade, R. By Communication, 1945.
(28) Philips, F. S., E. St John and A. Gilman. Unpublished observations
(29) Mc Namara, B. P. and F. Hopkins, Personal communication, 1945.
(4) Cameron, G. R. and F. Burgess. By Communication, 1943. Incl 2 to report of Investigation 5250 TIC 27 Mar 1947

II. A study on the manufacture of Benzolhexachloride.

YOSHIO MIKAMI

2 July 1946

A. Objective of the study.

The "DDT" used by the U.S. Army is a new powerful insecticide and its production stages are:

1. The manufacture of monochlorobenzeol.
2. The manufacture of chloral.
3. The manufacture of D.D.T. by the condensation of monochlorobenzol and chloral and treatment with CH_2SO_4 SO_3. It is later refined by crystalliz ation.

Technical skill and a great amount of time are required to make the chloral react chemically. These are the following defects:

1. The yield of D.D.T. after refining the crude D.D.T. is very low.
2. The chlorinating reaction is a substitute reaction and consequently only half of the chlorine is used in the process while the other half forms hydrochloric and (HCl).

GAMMEXANE investigated in ENGLAND as a substitute for D.D.T. is a compound of benzene with six atoms of chlorine attached. The chemicals used are benzene with sodium hydroxide as a catalyzer. All the chlorine reacts in a single reation. The crude form of the end product is obtained by filtering and no refining is necessary.

Its insecticide power is said to be greater than that of D.D.T. The only defect lies in its peculiar odour. This can be removed by washing or recrystallization.

In view of the above points, it was decided that benzol-hexachloride, rather than D.D.T., will be manufactured as an insecticide. Only the former has been investigated.

B. Details of the investigation.

There is little literature pertaining to the manufacture of benzolhexachloride (mathews, SOC 59, 165-1891, etc.). The methods described in books and documents are all the same the method is as follows.

Benzene is floated on a one per-cent solution of sodium hydroxide and chlorine is added under diffused light. As the chlorine is added, the benzene increases in specific gravity and precipitates. More benzene and chlorine is added. This mixture is allowed to stand until crystals form these are then separated from the solution. The oily filtrate mixture is used to obtain more crystals by treatment with new additions of the catalyser and chlorine.

With this above method, described in literature, the yield of benzolhexachloride is less than 1/500 of the benzine used in one charge by weight. A series or investigations has been out under various conditions and the following results were obtained.

1. The existence of light is absolutely necessary and short wave rays even more effective.
2. A temperature lower than ten degrees Centigrade is required for the reaction of chlorine.
3. When the sodium hydroxide solution (catalyser) is increased, the yield also increases.

The following results were obtained from the experiments.

Refer to trans. page 3 and 4.

EXPERI- MENT NO.	BENZENE	1% NaOH: CONDITIONS OF ADDING CATALYSER AND ITS SUPPLEMENTAL SUPPLY	CONDITION OF REACTION	TEMPERATURE AT WHICH RE-ACTION TOOK PLACE	HOURS REQUIRED FOR RE-ACTION	YIELD OF $C_6H_6Cl_6$	YIELD PER 1000GMs of
1	1) 100GMs 2) separated oil from above reaction 3)-11-	1) 300GMs. added as the initial charge. 2) 300 " 3) 300 "	under diffused light	1) rm. temp. on 30 Mar. 2) " 1 Apr. 3) " 2 Apr.	-	1)17GMS. 2) 8 " 3)15 "	1)17GMS. 2) 8 " 3)15 "
2	1) 100GMS. separated oily mix- 2)ture from above	1) 300GMS. After 2 hours reaction 3GMS. NaOH Supplemented. 2) 300GMS. added as initial charge.	"	1) " 2 Apr. 2) " 4 Apr.	4	1)45 " 2)17 "	1)45 " 2)17 "
3	1)100GMS. 2)oily mixture from above	1)300GMS. added as initial charge and 0.5GM. of benzol peroxide, simultaneously. 2)at water layer after above reaction, 3MGS. of NaOH supplemented.	"	1) " 6 Apr. 2) " 9 Apr.	1) 7 2) 4	1)19 " 2)9 "	1)19 " 2)9 "
4	100GMS.	300GMS.	in the dark	" 13-16 Apr.	7	1.5 "	1.5 "
5	1)500GMS. 2)oily mixture from above.	1)2,000GMS. after 5 hrs. reaction 20GMS. of NaOH supplemented 2)2,000GMS. added as the initial charge.	1) under diffused light 2)ultra-violet ray for 3 hrs.	1) " 18-19 Apr. 2) " 23 Apr.	1) 10 2) 7	1)130 " 2)37 "	1)26 " 2)7.4"
6.	1)500GMS. 2)oily mixture from above	1)2,000GMS. added as the initial charge. 2)2,000GMS. added as the initial charge.	1)ultra-violet ray for 2 hours 2)diffused light.	1) " 30 Apr. " 1 May 2) " 3 May	1) 7 2) 5	1)58 " 2)78+ 6 " crystal-ized	1)11.6 " 2)15.6 +1.2 "

Incl 5 To report Investi[illegible]ion 5250th T.I.C. 27 March 1947

7	100GMS	300 GMS. added as the initial charge 0.5GM. of benzolperoxide supplemented	in the dark	rm. temp. on 7-8 May	11	44GMS.	4GMS.
8	500GMS.	1,000GMS. each in both charges counter flow occurred. (TH-?)	diffused light	rm. temp. on 8 May	9	28GMS.	5.6GMS.
9	100GMS.	300GMS. added as initial charge and 1GM. of benzo-lperoxide supplemented	in the dark	rm. temp on 15 May	6	3''	3''
10	100GMS.	300GMS added as initial and 1GM. of ammonia-peroxide supplemented.	"	rm. temp. on 16-18 May	15	Trace	Trace
11	-	-	-	-		-	-
12	500GMS.	2,000GMS. added as initial charges and 20cc of 3% H_2O_2 supplemented.	diffused light	rm. temp. on 1-7 Jun.	32	43GMS.	8.6GMS. oily mixture 530GMS.
13	500GMS.	2,000GMS. added initial charge	"	12-18 Jun.	35	25 ''	5GMS.
14	100GMS	300GMS. added as initial charge.	"	5°-$15^{\circ}C$	5	28 ''	23'' 112''
15	500GMS.	2,000GMS. added as initial charge	"	10°-$15^{\circ}C$	12	37 ''	7.4''
16	100GMS.	300GMS. added as initial charge.	"	$5^{\circ}C$	2	34 ''	34'' 97''
17	100GMS.	watery layer of no. 16 used here again	"	$5^{\circ}C$	6	at gas phase wall 415GMS.	4.5''
18	100GMS.	300GMS. after 1 hour reaction, 3GMS. of NaOH supplemented	"	3°-$5^{\circ}C$	3	89''	89'' 81''
19	100GMS.	300GMS. after 1 hour reaction, 10cc of 3% H_2O_2 added	"	"	3	25''	25'' 113''
20	100GMS.	300GMS. after 1 hour, 3GMS. of NaOH, and after 2 hours further 3GMS. of NaOH supplemented	"	"	3	142''	142'' 7[illegible]''

C. Evaluation of experimental results.

1. Light is absolutely necessary for the chemical reaction. Experiments in the dark were unsuccessful as shown in experiments No. 4,7,9, and 10.

2. The yield can be increased by supplementing the sodium hydroxide solution, catalyser for the reaction as shown in experiments No. 2, 5, 18 and 20.

3. It is important to let the reaction take place at a temperature lower than ten degrees centigrade. Experiments No.'s 7 through 13, conducted at room temperature around May and later, resulted in yields of less than ten grams of benzolhexachloride. The yield can be increased by lowering the temperature as shown in experiments No.'s 14, 16, 18, 19 and 20.

4. The sodium hydroxide solution used once as a catalyser cannot be used for the next reaction as proved by experiment No. 17. (This conclusion reached after reviewing section 2).

EXTRACT

Osaka, February 12, 1947

CENTRAL LABORATORY, KANEGAFUCHI SPINNING CO., LTD.

123 Tomobuchicho, Miyakojimaku

Osaka, Japan.

SEMI-ANNUAL REPORT

For period July 1 to December 31, 1946.

1. Name of Research Laboratory or similiar organization:

Romaji: Chuo Kenkyusho, Kanegafuchi Boseki Kabushiki Kaisha.

English: Central Laboratory, Kanegafuchi Spinning Co., Ltd.

2. Address and Telephone Number of Laboratory or similiar organizations:

Osaka-shi, Miyakojima-ku, Tomobuchi-cho, 123.

Telephone: Horikawa 571-577, 1131-1132.

3. Ownership: Kanegafuchi Spinning Co., Ltd.

4. Name of Chief Research Engineer or Scientist:

Name: Yoshiichi Masuda.

5. Name of Key Personnel:

Name: Kaneki Noyori
Tatsusuke Hakoda
Miroru Yajima.

12. Research projects completed during the period covered by this report.

(1) DESIGNATION OF PROJECTS:

Research on the manufacturing of benzene-hexachloride.

Persons in charge:

Yoshio Mikami and Noboru Hayashi.

Detailed Reports:

(a) Purpose

For the prevention of typhus and euptive typhus, manufacturing of the insecticide which destroy fleas, lice ticks, ants, cockroaches and destructive insects of domesti animals and of agricultural products.

(b) Theory

Some benzene was placed upon the surface of a dilute solution of sodium hydroxide or sodium hypo-chlorite in a flask, a steam of chlorine was passed in the mixture, 3 Mol Chlorine was added to benzene and crystallized benzene-hexachloride. The reaction formula is next:

$$C6H6 + 3\ Cl2 = C6\ H6\ Cl6$$

(c) Experiments

When benzene-hexachloride was prepared by the method of Mauhews (Soc. 59, 165 (1891), some benzene was placed uo the surface of a dilute solution of sodium hydroxide in a flask, and a stream of chlorine was passed until benzene san to the bottom of the flask.

It was then corked up then placed outside the laboratory for the night, and the crystals of benzenehexachloride were separated from the oil and aqueous solution. But the yield secured in this process was not satisfied, moreover, the crystals could not be readily be separated from the oily matter in summer. We experimented on different kinds of these reacting conditions. Results shown in next table.

Benzene (g)	Catalyser		Reacting Condition		Product
	1% NaOH Soln.	Supplement of 10% NaOH Soln. (g)	Temperature (^{o}C)	Time (hr)	Yield of C6H6Cl6 (g)
100	300	0	Room Temp. of March 30	5	17
100	300	0	April 6	7	19
100	300	After 3 hrs. 30	April 2	4	45
100	400	0	May 1	7	11.6
100	400	0	June 1 - 7	43	8.6
100	400	0	June 12 - 18	25	5
100	300	0	5-15^{o}		
100	300	0	5^{o}	2	34
100	300	After 1 hr. 30	3-5^{o}	3	89
100	300	After 1 hr. 30 After 3 hrs. 30	3-5^{o}	3	142

(d) Results

The reaction which was acted chlorine in beneneze and separated benzene-hexachloride was exothermic reaction and was catalized by ClO' ions. Therefore cooling and existence of ClO' ions are necessary conditions of this reaction.

When benzene-heachloride was prepared by this method, the yield is nearly seven times that of the method of Mauhe in laboratory scale.

We set agoing model machine of preparation and then prepared benzene-hexachloride by passing a dilute chlorine gas which contained air for the factory production. Result shown in next table.

	Benzene	chlorine		NaOH	Average yields of C6H6Cl6
In Laboratory	100	Liquid Cl2		6	140
On Model Machine	100	" "	115	14	77
Factory production	100	dilute Cl2	242	17	50

(c) Conclusion

We improved the preparation method of benzene-hexachloride which had been experimented by Mattews. Namely, the reaction was acted in low temperature and the catalyser solution was dropped on the way of the reaction, consequently the yield was increased and would possible the industrial production. This method has been found to be no dangerous for preparation of benzene-hexachloride by passing a dilute chlorine gas which contained air.

13. Projects for Research on which active work planned during the next six month period:

1. *************
2. *************
3.

1. Designation of project:	Agricultural medicine.
2. Persons in charge:	Kaneki Noyori and Takashi Narakawa.
3. Purpose:	We are going to test, agri cultural insecticide, the efficacy as a substitute of petroleum emulsion.
4. Summary of manufacture:	Benzol hexachloride which is aroma hydrocarbon-chloraddition compound and para-toluenesulphon-chloride which sulphur compound, is mixed soluble in orthodichloro-benzol, i which add soluble a little Oleic acid and mixed colloidal clay "Ben onite" in water floating more than one weel, stirring mixed appropria alkali, become stable emulsion.

AN EXPLANATION OF D.D.T.

By SHISHIDO KEIICHI
(Professer of Engineering, KYOTO University)

Note: European names are inaccurate due to difficulty in transliteration.

It has been stated that there are now over 400,000 organic compounds, however this number is increasing yearly. We do not go out of our way to discover compounds in our research laboratories but even there, in the course of our studies, over a period of a months time one or two new compounds may be found. It is not unusual for a chemist who has specialized in the study of organic chemistry to find a new compound during some reaction. Usually, the chemist records the characteristic properties of the newly discovered compound without delving further into its potentialities. The fact that research into the properties of the newly discovered compounds must be done only by members of the chemical research profession is unfortunate. It would be advantageous if research along these same lines were performed by specialists in Medicine, Agriculture, Industry, Physics and other fields of definite specialization. There are people who are qualified in all of these fields, but as a rule, they are not as strong in those lines as a specialists would be. For this reason imperfect research is the rule rather than the exception. The present situation is that not only is there almost no consideration given to possible application of the newly discovered compound but there is little or no liaison between the discoverer and potential users. For these reasons the amount of organic compounds actually utilized numbers only 10,000.

Doctors and chemists cooperated in testing and evaluating various compounds while searching for an anti-malarial drug and other drugs. New compounds which occurred in the midst of a reaction during research have not been fully exploited. It was extremely fortunate that the SULFA compounds and HORMONES, which were discovered some ten years prior to their use, were finally utilized.

Original Synthesis

Mr ZEIDLER, a student of Professor BAYER, made DICHLORO DIPHENYL TRICHLORYL ETHANE in 1878 only as an example of the BAYER reaction. According to a publication by the German Chemical Association, this compound was described as being a white powdery substance with a melting point of 105 degrees C. The compound was not considered important enough to list in chemical manuals and it was not until 65 years later that a practical application was found.

The Discovery of Uses for the Compound

GEIGI, Co. in Switzerland, is one of the most famous chemical factories in the world. Dr. PAUL REVGER, director of this company thinking that a highly effective product would result from combination of CHLORAL, an anaesthetic and CHLORBENZOL, an insecticide, attempted to synthesize this compound for use as a powerful insecticide. He was successful and named it DDE. It then came to his attention that the compound had already been discovered, and he was unable to obtain patent rights. The raw materials and manufacturing procedure were simple. In 1939, this compound was sold under the name of GESAROL, NEOTITE, and other names, for use in foods, vegetables and against insects or flies. This company kept the formula of the compound a secret.

Use by the AMERICAN Army

A resident American Army officer realized the possibilities of GEZAROL and sent a sample to the United States. For some reason the German Government, cogniscant of the shipment to America took no steps toward stopping its delivery. The compound was diluted to 5% of its strength but after two months of study and analysis the main components were discovered. The Swiss Government also knew of the analysis being made in America. This was used as a counter-measure against Malaria which is more dreaded than enemy machine guns. The compound was accepted by the Army and was then made in large quantities. It played a major role in the war and was used in every phase from North Africa to Japan.

Trade Names

In the following paragraph, I will discuss the various names of this compound. If the method employed by the Germans is followed, the result will be DICHLORO DIPHENYL TRICHLORYL ETHANE. This name is too long and technical for the layman, so it has been shortened to D.D.T. The use of D.D.T. by the American Army is equivalent to several million dollars

worth of advertising, and the name "DDT" has become known to millions of people. We know of the events in America through TIME and NEWSWEEK and DDT has become a topic of everyday conversation.

Peacetime Use.

Since D.D.T. was not utilized by the American Army to kill men, it was used in a peaceful capacity and because of war it was produced on a large scale. According to the papers it is being used more and more in public sanitation and in the home. There is room for improvement in the method of spraying and dispensing DDT but it can be generally accepted that this is the method that must be employed. The use of DDT was hasty but it would appear that no mistake was made in its use.

The knowledge of the principles involved in use for agriculture is still imperfect. However, research is still going on in an attempt to find the amount to be used and poisonous properties. Since Japan is totally different from America so far as weather, insects and crops, etc. are concerned, we must investigate this matter thoroughly from our own standpoint.

Methods of Synthesis.

The most effective method economically for JAPAN to use at this time is that used by Mr. Zeidler which is simply the combination of CHLORAL and CHLORO BENZOL using SULPHURIC ACID.

A Detailed Explanation of the Synthesis

In the electrolysis of a solution of table salt in water, Caustic Soda, and a chloride by-product are formed. This chloride is combined with benzol which has been derived from coal tar. Chloro benzol is thus formed. Chloro benzol has the following properties:

1. Liquid in natural state
2. Mixes with and dissolves in water
3. Intermediate products
    a. Dye
    b. Medicine
    c. Dulcin (sugar substitute)

During the war chloro benzol was used as a source for picric acid and HEXYL (?) in making gunpowder. Since this was produced in great quantities during the war our country is well acquainted with the production methods.

Chloral is extracted from alcohol in the same manner that the chloride is derived from a salt-water solution. Chloral has the following properties:

1. Liquid in natural state.
2. In reaction with water forms a chloral hydrate, a crystal.

It is easier to handle in this state. Chloral is used to some extent in the manufacture of sleeping drugs and in the laboratory as a testing agent. This was manufactured on a small scale in our country. The process of extracting chloral from alcohol is a tedious and difficult one. Even though the cost of making medicine is using chloral is prohibitive, the fact that this medicine is used only in small amounts makes the cost of production possible.

Due to the fact that D.D.T. should be used in great quantity by all people this method of producing chloral makes it almost impossible for it to be used as the raw material for D.D.T. This method could not compete in the world of free enterprise. In the future chloral must be studied from an industrial standpoint. The difficulties of manufacturing D.D.T. exists in the production of chloral.

After the chloral has been extracted it is unnecessary to change it to chloral hydrate. Together with chlorobenzol the chloral will be put into concentrated suphuric acid and stirred at a normal temperature, from which the crystals of D.D.T. will be formed. The progress of this chemical reaction can be easily perceived. Since an iron container can be used to mix these chemicals the processing is made easier. After the completion of this reaction the crystalized material can be collected by filtration. Fuming sulphuric acid will be added to the diluted suphuric acid in order to concentrate it. This enables the processing of D.D.T. to be repeated. Although this is considered as one method of producing D.D.T., difficulties were met in obtaining materials to filter the sulphuric acid. A more economical method is to put the reacting mixture into a large quantity of water in order to dilute the sulphuric acid, then scoop out the D.D.T. which is not soluble in water and cast away the diluted sulphuric acid. In this process there are fewer difficulties.

Characteristics

D.D.T. is a white crystalline powder possessing some odor. It is not soluble in water but is soluble in organic solvents. It neither attracts nor repels insects. D.D.T. does not kill insects through their respiratory organs or their foods but through chance contact and once penetrating their bodies it attacks the nervous system. D.D.T. is believed to be effective for all insects whether they are in their matured or larval stage with the exception of coccidae (latin), plant lice and red ticks. The effectiveness on these last mentioned is doubtful as yet. Naturally D.D.T. which is poisonous to harmful insects also is poisonous to useful insects. However, this applies to all insecticides.

Comparatively speaking, D.D.T. is very slow in showing its effectiveness. Fleas and mosquitoes are killed in 30 minutes while bed bugs in a few hours, cockroaches a week after D.D.T. has been applied. For this reason even after using D.D.T. we are stung by mosquitoes. With the combination of D.D.T. and Pyrethrin, a component of pyrethrum, or pyrethrum powder both of which are effective but unreliable, the effectiveness will be increased. All insecticides produced from natural resources such as Pyrethrin and Rotenon (See Annex 2) are easily decomposed and made ineffective in a very short time through contact with sunlight, water or air. On the other hand D.D.T. is extremely stable and not easily decomposed. The lasting effectiveness of D.D.T. is one of its greatest qualities.

Although the exact lethal dose cannot be determined, it is said that 2.4 grs. of D.D.T. injected into a human, will be sufficient to kill him. From this we can gather that it is difficult for D.D.T. to poison humans. Particularcare is necessary to prevent the scattering of D.D.T. on foods. According to our experiences it is not harmful when it comes into contact with the human skin in powdered form. In liquid form it will penetrate the humane skin and prove very irritable. During experiments with liquid D.D.T. it proved irritable when it contacted the humane skin but when washed with soap and water this condition completely disappeared.

D.D.T. is some what harmful to melons and stunts the growth of tomatoes. Futher research is necessary to determine the reaction on agricultural products.

Application

When D.D.T. was produced in our laboratory we decided to test the effectiveness of D.D.T. on harmful insects found in water closets, Japanese mats etc. At present our country is suffering extreme shortages of organic solvents including petroleum. Due to this unavoidable situtation and to the construction of japanese houses, the powdered D.D.T. was thought to be more convenient. The effectiveness of D.D.T. was weakened ten percent by adding talc powder or Fuller's earth. After spreading D.D.T., flies in water closets were killed in several hours and fleas on Japanese mats were exterminated in a few days. When spread over the water surfaces of the emergency water tanks, mosquito larvaes were killed before they had a chance to mature.

According to an American magazine, when a petroleum solution containing five percent D.D.T. is sprayed over walls, ceilings and floors it is effective for six months against flies, mosquitoes and bed-bugs. The D.D.T. sprayed from airplanes appears to be this petroleum solution according to newspaper items. Water suspended D.D.T. and emulsions are also used.

Recently two or three companies in Japan started producing and selling D.D.T. At present the powdered form seems to sell the best. This is probably due to its cleaniness and convenience when applied for domestic use.

Similar compounds

Since D.D.T. was discovered, the insecticidical strength of chlorides was acknowledged. Anticipating other insecticides to be derived from these chlorides, research was carried out in various circles to discover similar compounds. The insecticidical strength of chlorides is probably due to the special state of atoms within the molecule. In the case of D.D.T. there is no doubt that the (chloride) group has an important role. One of the requirements necessary to strengthen the insecticide is that the standard amount of the chloride group should exist within the compounds. If the molecular weight of the becomes too great, the reacting strength is decreased, consequently it would not effect animals. On the other hand, if the

molecular weight is too small, the volatility of the compound is increased to the point where it will be dangerous to both humans and live stock. In addition, this compound will give forth a bad odor and the effective period will be shortened. Therefore, an organic chloride within the above-mentioned limits should be discovered. However, according to the usage, the limit should be changed. For example, in cases where volatile strength is especially required, chloropicrin will be used in spite of its danger to humans and live stock.

GAMMEXANE or (666), discovered by the English, Imperial Chemical IndustrialCo., at present, is regarded as an excellent insecticide and is derived from the above mentioned requirements. This insecticide is advertised as being five times more powerful than D.D.T. and is sometimes called "Super DDT". The formula, as in the case of D.D.T. is already known. The chemical name is BENZENE HEXACHLORIDE and not HEXACHLOROBENZENE. According to an old document BENZENE HEXACHLORIDE is made by passing chlorine gas into a solution of caustic soda with benzene floating upon the surface. Although the chemical formula for benzene hexachloride is quite different from D.D.T. it has the same appearence, that of a white crystalline powder. The manufacturing method used by Imperial Chemical Industrial Co., is not known. We experimented with the above-mentioned method, but the results were poor since only a small amount is benxene hexachloride resulted. The advantages of benxene hexachloride over D.D.T. are as follows:

1. Alcohol derived from food stuffs is not used as its raw material.

2. One chemical reaction is necessary. These advantages of BENZENE HEXACHLORIDE will provide a better prospect for future commerical production. After further research, a new method was discovered to obtain a large amount of BENZENE HEXACHLORIDE from a small amount of raw material. According to our tests BENZENE HEXACHLORIDE was proven definitely superior to D.D.T. However, its greater volatility makes its lasting effectiveness shorter. Its harmful qualities upon agricultural products is still uncertain.

Our tests showed that 7,5 per cent of BENZENE HEXACHLORIDE powder applied on water closet and JAPANESE mats was comparable to 10 percent of D.D.T. in its effectiveness. The bad odor from its volatility was not stronger than D.D.T. when the refined product was used.

Among the chlorides which fulfill the two requirements mentioned above, the molecular formation of BENZENE HEXACHLORIDE is the simplest. Its raw materials are easily obtained and its manufacturing process is easy. This makes it most suitable for the present condition of our country. It has its weak points but it also has its strong points to enable it to withstand future competition in the commercial market.

Although the volatile strength of the compound is not always proportionate to its molecular weight, its strength is roughly estimated through this method. The molecular weights of D.D.T. and BENZENE HEXACHLORIDE is 365 and 291 respectively. In the process of manufacturing BENZENE HEXACHLORIDE we used DICHLO-BENZENE as a substitute for the raw material, BENZENE, and produced DICHLOROBENZENE HEXACHLORIDE having the same distinctive appearence of white crystalline powder. Its molecular weight is 360 which is almost equal to D.D.T. This compound also is a good insecticide and has commercial prospects. The tests on its volatility and others have not been completed yet, due to lack of necessary equipment in our research laboratories. (Professor of KYOTO University doctor of engineering)

electroysis

$NaCl + H_2O$ ---------------- $NaOH + H_2$ --------- Cl_2

table salt, water, caustic soda, hydrogen, chlorine

benzene $+ Cl_2$ (chlorine) -------------- HCl + Cl chlorobenzene

CH_3CH_2OH (ethyl alcohol) ------ $4Cl_2$ (chlorine) ------------------ $5HCl$ (hydrochloric) -------- CCl_3CHO (chloral)

Cl

Cl Cl

Cl C--------CHO + 2 Cl------Cl C--------C---------H

Cl Chloral H_2SO_4 sulfuric acid Cl chlorobenzene

(catalyzer)

Cl D.D.T.

Cl Cl

H H

$3Cl_2$ --------Cl H H Cl $(C_6H_6Cl_6)$

H H

Cl Cl

benzene chlorine Benzene hexachloride

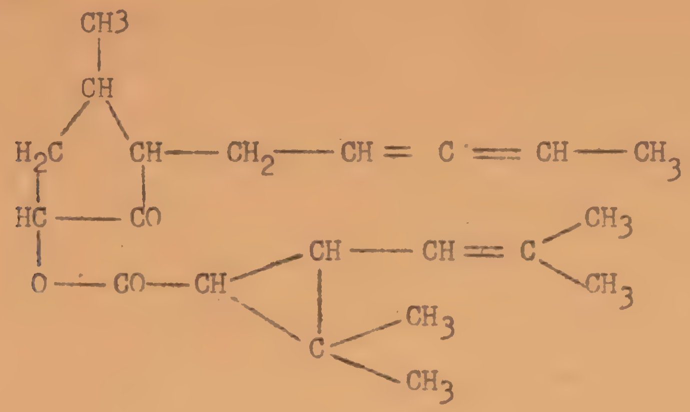
These chemical formulae are for the benfit of readers who have some knowledge of chemistry. Those readers who do not understand chemical foru[m] will have no difficulty in understanding this short article.

Annex 1 to Translation "An Explanation of D.D.T.

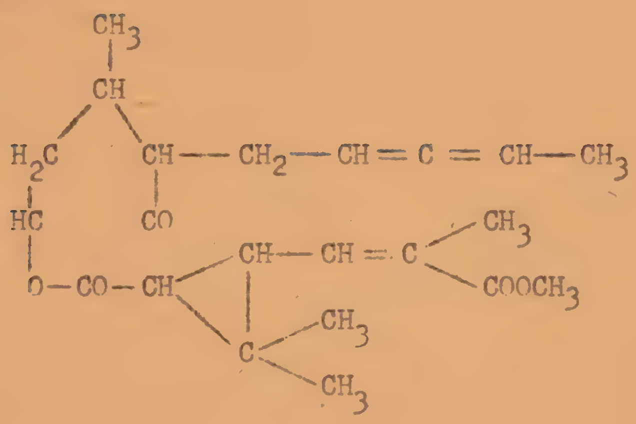
1. Pyrethrin
(Insektenvertilgungs mittel.)

a. Pyrethrum
The capitate flower of "Chrysanthemum cincerariifolim Bocc." or "chrysanthemum roseum" is picked and dried and pulverized, out of which is extracted the insecticidal substance. The chemical construction of this substance had two forms, Pyrethin 1, and Pyrethrin 2, according to Staudinger and Ruzick, 1924.

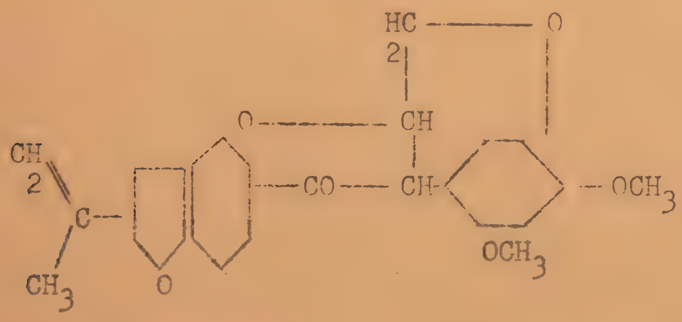
b. Pyrethrin 1



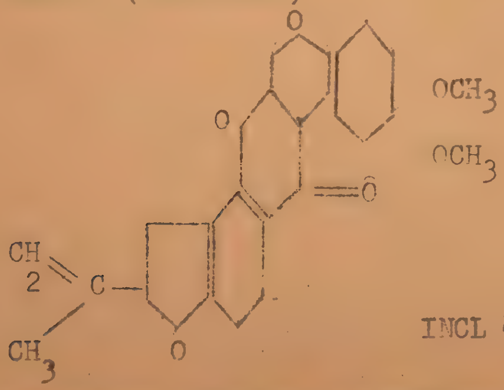
c. Pyrethrin 2



2. Rotenon
The insecticidal substance found in the root of Derris elltica Benth and Melletta taiwaniana Hayata (commonly known as a kind of wistaria) is called Rotenon. This is effective in killing fleas, lice, and insects harmful to agricultural plants. It is also used in curing the skin disease -scabies. This substance mixed in soap solution is on the market under the trade name of "Neoton".
The chemical construction of this substance is given as follows:
Form 1 (Takei, Miyajima and Ono)



Form 2 (Butenandt)



APPLICATION FOR PERMISSION OF MANU-
FACTURING AND SELLING OF "INSECTICIDE"

Location of plant: No. 125 Tomofuchi-cho
Miyakojima-ku, Osaka

Business office: The same location.

Location of godown: The same location.

The name of president: Shiro Kurachi.
Kanegafuchi Spinning Co., Ltd.

Birth date: Nov. 25, 1877

The name of goods Kanebo BHC

Utility: A. Insect powder for the prevention of epidemics.

B. Liquid insecticide for disinfection and agriculture.

Directions for use: Powder;
Spreading as the same method as DDT for extermination of lice, flea, mite, etc.

Liquid;
Liquid insecticide for disinfection and agriculture.

A. Insect-powder.

1. Method of manufacturing.

Some benzene was placed upon the surface of a dilate solution (about 1 per cent) of sodium hydroxide in a flask and a stream of chlorine was blew in the bottom of the mixed liquid. The benzene change in a heavy yellow oil and its precipitates in the bottom of the sodium hydroxide solution making a crystallise of benzenhexachloride.

After the crystal of benzene-hexachloride was crushed and mixed with Talk or Clay, the goods was manufactured.

2. Raw-material. (per ton)

Benzene-hexacloride	100 kg.
Talk or Clay	900 "
Goods	1,000 "

Remarks: Raw-material of benzene-hexachloride 100 kg. is as following:

Benzene	200 kg.
Chlorine	200 "
Canatic soda	25 "

Remarks: We can use a weak chlorine liquide mixed air for the purpose of disinfection of drinking water in Kanegafuchi Takasago Chemical Factory as chlorine.

3. The list of Kanebo B.H.C. Insect-powder.

Experiment.

The person in charge Kyoto Imperial University a doctor of science Mr. Tokuda.

The method of examination.

insect anaesthetized, puts in a test-tube contained a insect-powder, and we courts a time a insect goes to the death.

Incl 9 to report of Investigation 5250th
T.I.C. 27 Mar '47

Result of the experiment.

(1) Flea imago

BHC (10%) 20 minutes, 40 ", 90 ", 3 hours, 7 ", 12 ", 48 ", 60 "

______________________________death
______________________________death (the same test tube).

______________________death
______________________death (the same test-tube)

DDT (10%) ______________________________death

____death
(the same test-tube)

____________________________________death
____________________________________death
(the same test-tube)

- a male
- a female ______ narcotism ______ activity ______ emaciation

______ utmost emaciation

Put concurrently two heads in the same test-tube for experiment of effect.

(2) Mosquito

BHC (10%) 5 minutes, 10 ", 15 ", 20 ", 40 ", 60 ", 120 "

Anopheles
culex ______________________death
culex __________________________death
culex ____________________________death

DDT (10%)
culex ______________________________death
culex __________________________________death

(3) House Fly

10 minutes, 15 ", 20 ", [illegible] ", [illegible] ", [illegible] ", [illegible] hours, 15 "

BCH(10%) ____________________________death

DDT(10%) ____death

(4) The relation with patent.

(register)

The relation with BHC patent not application that to investigated by the patent office of Japanese Government.

(B) Liquid insecticide.

1. Compounding method.

I Mol. toluene acting at 15 -20 oC upon IV Mol. chloro-sulphuric acid and Benzene-hexachloride which was made in A (1). makes P-tolunene-sulphonylchloride.

Mix and melt this P-toluene-sulphonyl-chloride into O-dichloro-benzene, then add some Oleic acid or vegetable oil and for which gives chymification adding Colloid clay and alkali of proper quartity.

Details of compounding method.
Mix and melt Benzene-hexachloride 100 kg and P-toluene-sulphonyl-chloride 97.74 kg into O-dichloro-benzene 343.2 kg and add Oleic acid 50 kg, then it makes the liquid A. Melt Bentnite 30 kg into water 359.76 kg and when let take its own course more than a week it makes the liquid B. Then, mixture A and B makes chymification liquid after adding and stiring Caustic soda 19,3 kg. (manufactures shows one ton)

2. Materials for mfg.

Benzene hexachloride	100 kg
P-toluene-sulphonyl-chloride	97.74
O-dichloro-benzene	343.2
Oleic acid	50.
Caustic soda	19.3
Bentnite	30.
Water	359.76
Itemization	
Benzene hexachloride	100 kg
meeds A (2) as the materials	
P-toluene-sulphonyl-chloride	100 kg
consists of toluene	200 "
& chlorosulphuric acid	800 "
O-dichloro-benzene	100 "
consists of benzene	499 "
chlorine	500 "
Alumium chloride	10 "

Remarks:

O-dichlorobenzene is the liquid which was reparted from Dichloro-benzene after exception of Monochloro-benzene from chloridized distiled Benzene and Cooling treatment of remained liquid. And this O-dichloro-benzene is the mixtures of Para-dichloro-benzene and Ortho-dichloro-benzene which has Ortho-dichloro-benzene as its ingredient.

The specific gravity of O-dichloro-benzene is 1.30

THE INVESTIGATION OF KANEBO BHC EMULSION EFFICIENCY

The Agricultural experiment station
of
Hyogo Prefecture,
Section of insect pathology.

Compound method.

The spread of soybeen casein 30 monme was disolved with water one to 18 litres, that put into the Kanebo BHC Emulsion and to make the dilute solution of same concentration. It to stir up used investigation method of medicine spread.
The investigation in the room that spread to insect body by simple spray or it put into the medicine solution and withdrew from the solution to adheres.
The field investigation that spread to insect body by simple spray.
Record of investigation.

(1) The larva of lady-bird

The first investigation on October 15, 1946.
The second investigation on October 18, 1946.

(Investigated in the room)

Test Division	First Test			Second Test			
	Number of Test Insect	Number of Death Insect	Percentage of Death Insect	Number of Test Insect	Number of Test Insect	Number of Death Insect	Percentage of Average Death Insect
1 Division BHC emulsion 30 times	10	7	70%	10	9	90%	80%
2 Division BHC " 50 times	10	6	60	10	8	80	70
3 Division BHC " 70 times	10	7	70	10	9	90	80
4 Division BHC " 100 times	10	2	20	10	6	60	40
5 Division Standard (Not spread)	10	0	0	10	0	0	0

Notice; The investigated death or not under the insect put into the medicine death insect investigation have done on the two days.

(2) The cockroach (Investigation on October 3, 1946)

(Investigated in the room)

Test division	Number of test insect	Number of death insect	Percentage of death insect
1. Division BHC emulsion 30 times	20	20	100%
2. -do- 50 "	20	20	100
3. -do- 70 "	20	20	100
4. -do- 100 "	20	20	100
5. Division standard (Not spread)	0	0	0

Notice: The medicine spread to the body of insect by simple spray death insect investigation have done on the one day.

(3) Medicine investigation, The garden radish (shogoin)
Investigated on October 16, 1946,
(Field investigation)

Test Division	Without of medicine damage	Degree of medicine damage	Growing condition after that
1. Division BHC emulsion 30 times	Exist	Some leaves would be death	The growing condition would be late one week as compared with standard division and the root growing no good.
2. -ditto- 50 "	"	many	Slightly as compared with 1 division
3. -ditto- 70 "	none	middle	Same condition with standard division
4. -ditto- 100 "	"	-	
5. Division, Standard, (not spread)	"	-	

Notice: To spread by the semi-automatic spray, medicine damages investigation have done on the one day.

(4) Medicine damage investigation, The rice-plant (Asahi)
Investigated on October 14, 1946 (Test in the pot)

Test division	Number of All leaves	Number of All stalks	Number of damage leaves	Number of damage stalks	Degree of damage	Remarks
1. Division, BHC emulsion 30 times	24	8	24	8	many	After the spread that growing condition no good The medicine of leaves has first become brown and the change inner gray to red spot
2. -ditto- 50 times	34	9	0	0	-	
3. -ditto- 70 times	25	11	0	0	-	
4. -ditto- 100 times	24	9	0	0	-	
5. Division standard (not spread)	34	8	0	0	-	

(5) Medicine Damage Investigation.

The Rice-plant (Asahi)

Investigated on October 3, 1946

(First Field Investigation)

Test Division	Number of All leaves	Number of All Stalks	Number of damage leaves	Number of damage Stalks	Degree of damage	Remarks
1. Division Emulsion 30 times	116	33	116	32	many	After the spread that growing conditions no good
2. Ditto- 50 times	not legible	23	74	12	slightly	
3. Ditto 70 times	not legible	not legible	not legible	not legible	not legible	
4. Ditto 100 times	not legible	not legible	not legible	not legible	not legible	
5. Division Standard (Not spread)	not legible	not legible	not legible	not legible	not legible	

Incl 9 to report of Investigation 5250th TIC 27 Mar '47

(6) Medicine Damage Investigation.

The Rice-plant (Asahi)

Investigated on October 4, 1946

(second field investigation)

Test Division (Prescription of Medicine)	Number of All leaves	Number of All Stalks	Number of damage leaves	Number of damage Stalks	Degree of Damage	Remarks
1. Division BHC emulsion 30 times, water 1 to soy-bean case in 30 momme	94	26	94	26	many	
2. " 50	71	20	53	2	slightly	
3. " 70	103	29	0	0	-	
4. " 100	86	25	0	0	-	
5. Division Standard (not spread)	104	30	0	0	-	